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- (71) Applicant (for AE, AG, AU, BB, BZ, CA, CY, GB, GD, GH, GM, IE, IL, KE, LC, LK, LS, MN, MW, NZ, SD, SG, SI, SZ, TT, TZ, UG, ZA, ZW only): **UNILEVER PLC** [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).
- (71) Applicant (for AL, AM, AT, AZ, BA, BE, BF, BG, BJ, BR, BY, CF, CG, CH, CI, CM, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, FR, GA, GE, GN, GQ, GR, GW, HR, HU, ID, IS, IT, JP, KG, KP, KR, KZ, LR, LT, LU, LV, MA, MC, MD, MG, MK, ML, MR, MX, MZ, NE, NL, NO, PH, PL, PT, RO, RU, SE, SI, SK, SN, TD, TG, TJ, TM, TR, UA, UZ, VN, YU only): **UNILEVER NV** [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).
- (71) Applicant (for IN only): **HINDUSTAN LEVER LIMITED** [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, 400 020 Mumbai (IN).
- (72) Inventors: **EMERY, William, Derek**; 64 Plymyard Avenue, Bromborough, Wirral, Merseyside CH62 6BP (GB). **GROOT, Andreas, Theodorus**; Unilever Research Vlaardingen, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).
- (74) Agents: **AGNEW, Andrew** et al.; Unilever PLC, Patent Dept., Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB).
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(54) Title: PRODUCTION OF ANIONIC SURFACTANT GRANULES BY IN SITU NEUTRALISATION

(57) Abstract: A process for the production of detergent particles, comprises feeding an acid precursor of an anionic surfactant and a neutralising agent into a horizontal thin-film evaporator/drier comprising a mixing region, a drying region and a cooling region. The resultant detergent particles are graded to separate an oversize granule fraction in which at least 70 wt% of the particles have a minimum diameter of 1000 µm. This fraction is fed back into the process.

- 1 -

PRODUCTION OF ANIONIC SURFACTANT GRANULES BY IN SITU
NEUTRALISATION

5 Technical Field

The present invention relates to a process for the production of anionic detergent particles and detergent compositions containing them. More particularly the present
10 invention relates to a process for the production of detergent particles having a high level of anionic surfactant which involves *in situ* neutralisation of an acid precursor of the anionic surfactant and drying of the surfactant thereby produced.

15

Background & Prior Art

Granulation processes involving mixing and agglomeration to form detergent granules have been widely investigated as
20 alternatives to spray drying processes. Such mechanical mixing processes offer a number of advantages over spray drying, e.g. production of higher bulk density products and increased formulation flexibility. These mechanical processes generally utilise technologies which are specific
25 to the particular type of mixing apparatus being used. However, most of them involve forming the salt form of anionic surfactants by neutralising the acid form of the anionic surfactant with an alkaline neutralising agent (usually in solid form), inside the mixing apparatus. This
30 is normally referred to as *in situ* neutralisation.

Although there are several types of mixing apparatus which may be employed, one common arrangement is a combination of

- 2 -

a high speed mixer/densifier followed by a moderate speed mixer/densifier, e.g. as disclosed in EP-A-0 420 317.

Another common arrangement was a so-called fluid bed
5 granulator, e.g. as described in WO-A-98/58046.

However, the present invention is concerned with a different class of apparatus, namely a horizontal thin-film evaporator/drier. Specifically, it is known that detergent
10 particles having high anionic surfactant levels can be prepared by processes in which acid precursors of anionic surfactants are neutralised with a neutralising agent in horizontal thin-film evaporator/drier (WO-A-96/06916, WO-A-96/06917 & WO-A-97/32002; WO-A-98/38278 & WO-A-98/40461) and
15 the mass is granulated and dried. As used herein, the term thin-film evaporator/drier is understood to include flash-driers and scraped-surface driers as described in WO-A-96/06916, WO-A-96/06917 & WO-A-97/32002.

20 Basically, a thin-film evaporator/drier comprises a cylindrical chamber in which is located a coaxial shaft on which is mounted a plurality of blade-like tools. The pitch of these tools can be set to different angles along the length of the cylindrical chamber, from input end to output
25 end. The clearance between the tips of the blade-like tools and the internal surface of the cylindrical chamber is very small, typically 5mm or less. The cylindrical chamber comprises at least a mixing region at or towards the input end of the cylindrical chamber, a cooling region at or
30 towards the output end of the cylindrical chamber and a drying region between the mixing and cooling regions. The drying region typically comprises one or more heating zones and the cooling region may comprise one or more cooling zones (although usually only one cooling zone. Each of the
35 heating and cooling zones is defined by a respective jacket

- 3 -

around the cylindrical chamber with a respective axial gap between each and through which jackets, a heating or cooling liquid, as appropriate, is pumped during operation of the process. The layering agent is typically an
5 aluminosilicate or a silica.

WO 97/32002 discloses a dry-neutralisation process in which high anionic surfactant-content detergent particles are manufactured by contacting a pumpable acid precursor
10 with a solid neutralising agent, such as for example sodium carbonate, in a thin-film evaporator/drier.

EP 555 622 (Procter & Gamble) describes the manufacture of detergent particles comprising anionic surfactant in which
15 acid precursors are neutralised in a high shear mixer by a stoichiometric excess of particulate neutralising agent, preferably sodium carbonate. The neutralisation reaction is optimised by using neutralising agent of a narrowly defined particle size range, namely 50% by volume has a particle
20 diameter of less than 5 microns.

EP 555 622 does not however relate to the production of high anionic surfactant-content detergent particles or to the use of thin-film evaporator/driers. According to the teaching
25 of EP 555 622, the neutralising agent is preferably present in an amount of at least five times that required for stoichiometric neutralisation. In addition, the ratio of liquid ingredients (e.g. acid precursor) to powder ingredients (e.g. neutralising agent) introduced into the
30 high shear mixer is most preferably from 1:2 to 1:3. It is not possible to make high anionic surfactant-content detergent particles using such excesses of neutralising agent and/or liquid to solid ratios. Indeed, the detergent particles of the examples contain merely 23 wt% anionic
35 surfactant.

- 4 -

All such technologies produce a product with a distribution of different particle sizes. Those which are very small are sometimes called ~~xxx~~ fines ~~xxx~~. It is common to recycle the
5 fines to the input stage of the mixer, in order to avoid having a dusty product. This is well known for various types of mixer. However, large particles, sometimes called ~~xxx~~ oversize ~~xxx~~, present more of a problem. Their presence in the product is undesirable, e.g. because they give rise to
10 negative consumer perception of product quality. In other mixing processes, e.g. as described in EP-A-0 420 317 and WO-A-98/58046, it is found that oversize particles cannot be recycled without having a deleterious on the product. This means that the oversize particles have to be milled prior to
15 recycling, which adds to production cost.

Surprisingly, it has now been found that in the case of processes utilising a horizontal thin-film evaporator/drier, oversize particles can be recycled without significantly
20 detracting from product quality.

Summary of the Invention

25 Accordingly, a first aspect of the present invention provides a process for the production of detergent particles, the process comprising feeding an acid precursor of an anionic surfactant and a neutralising agent into a horizontal thin-film evaporator/drier comprising a mixing
30 region, a drying region and a cooling region, removing the detergent particles so formed from an outlet of the evaporator/drier, separating an oversize granule fraction in which at least 70 wt% of the particles have a minimum diameter of 1000 μ m from the detergent particles and

- 5 -

recycling the oversize granule fraction to be fed back into the thin film evaporator/drier.

5 This invention also provides detergent particles obtained by the process.

Detailed Description of the Invention

The Oversize Granule Fraction

10

The oversize granule fraction comprises at least 70 wt% of particles having a minimum diameter of 1000 μ m. However, preferably at least 95 wt% of particles in the oversize fraction have a minimum diameter of 700 μ m. Particles having a minimum specified particle diameter means those particles which would be retained on a sieve having a mesh size corresponding to that size.

20 Preferably, at least some of the recycled granule fraction is fed back into the evaporator/drier before the cooling region, i.e. into the mixing region and/or onto the drying region and/or between those two regions.

25 Preferably also, the recycled oversize granule fraction constitutes an average of from 1% to 50%, more preferably from 5% to 25% by weight of the total solids dosed into the evaporator/drier.

30 Simultaneous neutralisation, drying and granulation process in a thin-film evaporator/drier

The process is carried out in a horizontal thin-film evaporator/drier (hereinafter referred to as an
35 ~~evaporator/drier~~). A commercial scale machine typically

- 6 -

comprises at least 300, preferably at least 500, more preferably at least 750, especially at least 1000 blade-like tools. The clearance between the blades and the internal wall of the chamber is suitably less than 20mm, e.g. 15mm or less, or even 10mm or less. The blade tip speed in operation is suitably $\geq 15\text{ms}^{-1}$, preferably $\geq 20\text{ms}^{-1}$. The ratio of exposed blade length to shaft radius is preferably less than 1, e.g. less than 0.5. Preferably, the large number of blades and the pitch of the blades also means that at least 40%, for example at least 45%, and even substantially the whole chamber wall (that part along the length of the shaft which carries the blades) is scraped during operation.

Initial mixing of the components occurs in the mixing region and the neutralisation reaction is begun. Mixing and neutralisation then continue throughout the process, through the drying and cooling regions.

The anionic surfactant acid precursor (hereinafter referred to as the ~~acid precursor~~) and neutralising agent are normally fed into the mixing region of the evaporator/drier. However, all or part of either component can be dosed into the drying region. Neutralisation occurs to form a surfactant paste, which is then converted into detergent particles by the drying and mechanical action of the evaporator/drier. The evaporator/drier exerts its drying action by forming a thin layer of material on a heated surface within the drying region.

The acid precursor is suitably fed into the evaporator/drier in a liquid phase. As acid precursors can be unstable, the neutralisation preferably occurs sufficiently rapidly and substantially completely such that thermal decomposition of the acid due to elevated temperature is minimised and desirably avoided.

- 7 -

The neutralising agent is introduced into the evaporator/drier as a solid particulate material.

Preferably, the amount of neutralising agent with respect to the acid precursor added to the mixing region is at least in stoichiometric equality, most preferably in excess.

Preferably, at least 1.25 times required for stoichiometric neutralisation is used but preferably no more than 2 times. Higher amounts of neutralising agent, e.g. no more than 5 times, no more than 4.5 times, or no more than 4 times that required for stoichiometric neutralisation can be used but these higher amounts are generally less preferred.

The acid precursor and neutralising agent may be added as a single stream to the evaporator/drier, or as two or more streams.

The drying region of the evaporator/drier basically comprises a tube which is substantially circular in cross section and is thus defined by a cylindrical wall. The material entering the drying region is heated. Typically this is achieved by heating the wall of the drying region by means of a heating jacket through which water, steam or oil may be fed.

The drying region may be divided into a number of heating zones, each heated to the same or a different temperature, preferably by means of a respective heating jacket. The temperature in the drying region is preferably maintained at at least 100°C, more preferably at at least 120°C, yet more preferably at at least 130°C. Higher temperatures are possible, but it will be understood by the skilled person that it is preferable not to exceed the thermal decomposition temperature of the acid precursor or the anionic surfactant formed therefrom. Depending on the

- 8 -

detergent active, temperatures up to 170°C or even up to 180°C are employed.

It has been found to be highly advantageous in the process of the invention to pass the material leaving the drying region of the evaporator/drier through a cooling region. The cooling region may be provided by a separate piece of apparatus, such as for example a cooling fluid bed, an airlift alternatively, may form part of the evaporator/drier apparatus.

The cooling region is preferably operated at a temperature not in excess of 50°C and more preferably not in excess of temperature 40°C, e.g. 30°C. Actively cooling the particles reduces the possibility of thermal decomposition occurring due to particles being heated to a high temperature. In addition, actively cooling reduces the risk of particles sticking/clumping which may occur when heated particles are allowed to cool passively.

Preferably, the cooling region is defined by a cylindrical wall which is cooled, for example, by a cooling jacket. Where the process is continuous, the evaporator/drier and the cooling region are suitably arranged so that the drying region and cooling region are substantially horizontally aligned to facilitate efficient drying, cooling and transport of the material through the drying region and cooling region in a generally horizontal direction.

In a preferred embodiment, the evaporator/drier apparatus includes the cooling region positioned after the drying region, and the cooling region is a tube which is substantially circular in cross section and is thus defined by a cylindrical wall. When such evaporator/drier apparatus is employed, a suitable temperature gradient is set up going

- 9 -

from, for example, at least 100°C at the inlet end to, for example, not more than 80°C at the outlet end.

Agitation of the materials in the drying region generally provides efficient heat transfer and facilitates removal of water. Agitation reduces the contact time between the materials and the wall of the drying region, which, together with efficient heat transfer, reduces the likelihood of ~~hot spots~~ forming which may lead to thermal decomposition. Moreover, improved drying is secured thus allowing a shorter residence time and increased throughput in the heating zone(s).

Preferably, the cooling region is also provided with agitation means to effect efficient cooling of the material therein. This may be a fluidising gas in a cooling fluid bed. Alternatively, where the cooling region is part of the evaporator/drier apparatus, it is preferred to use the same rotating agitation means as defined above in relation to the drying region.

It will be understood that the cooling region may comprise more than one cooling zone.

In a preferred embodiment, drying and cooling regions together comprise three zones defined by a cylindrical wall, the first two zones being heating zones defining the drying region, and the third zone being the cooling region. Acid precursor and neutralising agent are fed in to the first zone and rotating agitation means comprising a series of radially extending paddles and/or blades mounted on a axially mounted rotatable shaft agitates and transports material through the heating and cooling zones to produce detergent particles.

- 10 -

Preferably, the evaporator/drier is operated at atmospheric pressure in counter-current or co-current with a gas stream at a throughput rate of 10-150 m³ per hour. The gas stream may simply be air, which may have been dried so as to reduce its moisture content, or may be a gas stream having an alkaline pH, such as for example a mixture of ammonia and air.

The process of the invention is preferably continuous as this facilitate continuous transportation of the particles.

Suitably the total average residence time in the drying region is from 30 seconds to 15 minutes, preferably from 1 minute to 12 minutes, more preferably from 2 minutes to 8 minutes. The average residence time may be determined by injecting a coloured tracer and plotting a concentration profile for the tracer exiting the drying region. The average residence time is taken as the value corresponding to 50% of the total area under the curve. Preferably, the measurement is repeated a suitable number of times.

The process of the invention may be carried out in any suitable apparatus. Suitable thin-film evaporator/drier apparatus include the ❖ Flash Dryer/Reactor❖ manufactured by VRV, the ❖ Turbodryer❖ manufactured by VOMM and a similar machine available from Bipex Hosokawa.

30 Acid Precursors

Prior to neutralisation and drying, the anionic surfactants are present and fed into the drying region of the evaporator/drier in their acid precursor form. The acid precursor can either be fed as an aqueous preparation or in

- 11 -

anhydrous form. If added as an aqueous preparation, it is preferred that the water content does not exceed 25% by weight, more preferably not exceeding 10% by weight.

5 Suitable acid precursors include:

- 10 - linear alkyl benzene sulphonic acids (LAS acids) which give linear alkyl benzene sulphonates (LAS) upon neutralisation. Preferably, any LAS anionic surfactant has a chain length of C_{8-18} , more preferably C_{9-15} and most preferably C_{10-14} .
- 15 - alkyl and/or alkenyl sulphuric acid half-esters (i.e. the sulphation products of primary alcohols) which give alkyl and/or alkenyl sulphates upon neutralisation. The present invention has especial applicability in the production of detergent particles comprising PAS having a chain length of C_{10-22} , preferably C_{12-14} ; Coco PAS is particularly desirable.
- 20 - carboxylic acids which give soaps upon neutralisation. Preferred carboxylic acids are fatty acids with 12-18 carbon atoms, such as for example fatty acids of coconut oil, palm oil, palm kernel and tallow.

25 Other suitable acid precursors include alphaolefin sulphonic acids, internal olefin sulphonic acids, fatty acid ester sulphonic acids and primary sulphonic acids.

30 It is also possible to use combinations of various acid precursors as will be apparent to the skilled person.

- 12 -

Neutralising agent

- The neutralising agent is a particulate base material capable of neutralising the acid precursor. In principle, any alkaline inorganic material can be used for the neutralisation but water-soluble alkaline inorganic materials are preferred.
- 5 Suitable neutralising agents include any salts of hydroxides, carbonates, bicarbonates and silicates. Suitably, the sodium, potassium, calcium or magnesium salts may be used. However, the sodium salt is preferred.
- 10 A preferred neutralising agent is sodium carbonate alone or in combination with one or more other water-soluble inorganic materials, for example, sodium bicarbonate or silicate.
- 15 Preferably, the neutralising agent has a D50 mean particle size of less than $40\mu\text{m}$, preferably less than $30\mu\text{m}$, more preferably less than $20\mu\text{m}$. As used herein, the term D50 refers to the value of particle size corresponding to 50% weight percent of the particles on a size distribution curve
- 20 (i.e. half of the area under the curve is to the right of this value, and half to the left).
- 25

Detergent particles

30

In addition to the acid precursor and neutralising agent, other liquid and solid components may be fed to the drying region of the evaporator/drier, and/or the cooling zone if present. For example, pre-neutralised surfactants, e.g.

- 13 -

PAS, LAS and LES may be fed into the drying region as separate streams and/or as an admixture with the neutralising agent and/or acid precursor.

- 5 However, it is desirable that the ratio of the total liquid ingredients to the total solid ingredients fed to the drying region of the evaporator/drier, or, where a cooling zone is employed, to the drying zone and cooling zone, is in the range 2:1 to 6:1, preferably from 3:1 to 5:1.

10

The detergent particles have an anionic surfactant content of at least 40% by weight. The present process can be used to make detergent particles with a anionic surfactant content of at least 50%, 60% or 70% by weight. The maximum amount is typically 90%, preferably 85% by weight.

15

It is desirable that the particles also comprise water in an amount of 0 to 8% and preferably 0 to 4% by weight of the particles.

20

Other non-surfactant components which may be present in the detergent particles include dispersion aids, preferably polymeric dispersion aids and more preferably urea, sugars, polyaklyleneoxides; and builders as hereinafter described.

25

If desired the detergent particles may comprise an organic and/or inorganic salt, e.g. a hydratable salt. Suitable materials in salts, preferably sodium, of tripolyphosphate, citrates, carbonates, sulphates, chlorides.

30

Aluminosilicates, clays, silicas and other inorganic materials may also be included.

35

The particles may also contain one or more nonionic surfactants, for example as mentioned below in the context of a base powder with which the particles are admixed.

- 14 -

Similarly, organic materials, e.g. PEG and other polymer builder or soap may also be included in the particles, also as mentioned below in the latter context.

5

It is especially preferred that a salt be present in the particle when the anionic surfactant component comprises LAS.

10 The salt may be present at a level of up to 50% and preferably up to 30% by weight of the particles.

Desirably the detergent particles have an aspect ratio not in excess of 2 and more preferably are generally spherical
15 in order to reduce segregation from other particles in a formulated detergent composition and to enhance the visual appearance of the powder.

Layering

20

The detergent particles can be coated by adding a layering agent either at the output end of the drying region of the evaporator/drier, in the cooling region, or in an additional step, e.g. on a vibrating conveyer belt. It is particularly
25 preferred to add layering agent in the cooling region.

The layering agent may be any material capable of coating the particles in order to improve the granularity thereof. Relatively inert materials are preferred for this purpose
30 but especially any of those inert materials which have a beneficial effect in the wash liquor, for example, aluminosilicates, silicas talcs and clays. A mixture of such materials may be used. Examples of aluminosilicates and silicas are outlined in more detail hereinbelow. The
35 presence of any such material as a coating on the finished

- 15 -

particles does not preclude the presence also of the material within the body of the particles.

In the case of aluminosilicates, these may be partly or
5 solely dosed earlier in the process, i.e. not completely, or
not at all, as layering agents. A particularly suitable way
of implementing this is to feed an acid precursor of an
anionic surfactant, a neutralising agent and aluminosilicate
detergency builder into the horizontal thin-film
10 evaporator/drier comprising a mixing region, a drying region
and a cooling region, to effect neutralisation of the acid
precursor, granulation, drying and cooling, to form the said
detergent particles, wherein at least some of the
aluminosilicate builder is fed into the thin-film
15 evaporator/drier between the drying region and the cooling
region and/or into the cooling region. Optionally, when all
of the aluminosilicate builder is fed into the cooling
region, the aluminosilicate is in an amount of more than 20%
by weight of the particles exiting the cooling region.

20

Relative to the total other materials of the particles, the
dosing weight ratio of the layering materials, e.g. added in
the cooling zone, is preferably from 1:3 to 1:20, more
25 preferably from 1:9 to 1:20.

Detergent compositions

30 The detergent particles may be post-dosed directly to a base
powder obtained from any conventional detergent production
process including a non tower process in which the
components of the detergent composition are mixed and
granulated as described e.g. in EP-A-367 339 and a spray
35 drying process optionally followed by a post tower

- 16 -

densification. As the detergent particles produced by the present invention may be post-dosed to such powders a significant degree of formulation flexibility is obtained and the level of active material in the fully formulated composition may be very high as desired. A further advantage is that a base powder which is substantially free of detergent active compounds may be produced as the detergent active compounds may be introduced substantially wholly as post-dosed particles.

10 The option of reducing the level of detergent active material in a base powder is especially advantageous where the base powder is produced by a spray drying process as a lower level of detergent active compound in the spray drying process permits a higher throughput to be secured thus increasing overall production efficiency.

20 Compositions according to the present invention may also contain, in addition to the detergent-active compound, a detergency builder and optionally bleaching components and other active ingredients to enhance performance and properties.

25 Detergent compositions of the invention may contain, in addition to the post-dosed detergent particles, one or more detergent-active compounds (surfactants) which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in ~~the~~ Surface-Active Agents and Detergents~~ed~~, Volumes I and II, by Schwartz, Perry and Berch. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

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- 17 -

Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C8-C15; primary and secondary alkyl
5 sulphates, particularly C12-C15 primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

- 10 Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₈-C₂₀ aliphatic alcohols ethoxylated with an average of from 1 to 20 moles ethylene oxide per mole of alcohol, and more especially the C₁₀-C₁₅ primary and secondary aliphatic
15 alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).
- 20 The total amount of surfactant present in the detergent composition is suitably from 5 to 40 wt% although amounts outside this range may be employed as desired.

- The detergent compositions of the invention generally also
25 contain a detergency builder. The total amount of detergency builder in the compositions is suitably from 10 to 80 wt%, preferably from 15 to 60 wt%. The builder may be present in an adjunct with other components or, if desired, separate builder particles containing one or more builder
30 materials may be employed.

Inorganic builders that may be present include sodium carbonate, if desired in combination with a crystallisation seed for calcium carbonate, as disclosed in

- 18 -

GB-A-1 437 950; crystalline and amorphous aluminosilicates, for example zeolites as disclosed in GB-A-1 473 201; amorphous aluminosilicates as disclosed in GB-A-1 473 202; and mixed crystalline/amorphous aluminosilicates as
5 disclosed in GB 1 470 250; and layered silicates as disclosed in EP-B-164 514. Inorganic phosphate builders, for example, sodium, orthophosphate, pyrophosphate and tripolyphosphate, may also be present, but on environmental grounds those are no longer preferred.

10

Aluminosilicates, whether used as layering agents and/or incorporated in the bulk of the particles may suitably be present in a total amount of from 10 to 60 wt% and preferably an amount of from 15 to 50 wt%. The zeolite used
15 in most commercial particulate detergent compositions is zeolite A. Advantageously, however, maximum aluminium zeolite P (zeolite MAP) described and claimed in EP-A-384 070 may be used. Zeolite MAP is an alkali metal aluminosilicate of the P type having a silicon to aluminium
20 ratio not exceeding 1.33, preferably not exceeding 1.15, and more preferably not exceeding 1.07.

Organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers,
25 and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and
30 succinates; and sulphonated fatty acid salts. A copolymer of maleic acid, acrylic acid and vinyl acetate is especially preferred as it is biodegradable and thus environmentally desirable. This list is not intended to be exhaustive.

- 19 -

Especially preferred organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%,
5 preferably from 1 to 10 wt%. The builder is preferably present in alkali metal salt, especially sodium salt, form.

Suitably the builder system comprises a crystalline layered silicate, for example, SKS-6 ex Hoechst, a zeolite, for
10 example, zeolite A and optionally an alkali metal citrate.

Detergent compositions according to the invention may also contain a bleach system, desirably a peroxy bleach compound, for example, an inorganic persalt or organic peroxyacid,
15 capable of yielding hydrogen peroxide in aqueous solution. The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. An especially preferred bleach system comprises a peroxy bleach compound (preferably
20 sodium percarbonate optionally together with a bleach activator), and a transition metal bleach catalyst as described and claimed in EP 458 397A and EP-A-509 787.

The compositions of the invention may contain alkali metal, preferably sodium, carbonate, in order to increase
25 detergency and ease processing. Sodium carbonate may suitably be present in an amount from 1 to 60 wt%, preferably from 2 to 40 wt%. However, compositions containing little or no sodium carbonate are also within the
30 scope of the invention.

Powder flow may be improved by the incorporation of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or

- 20 -

acrylate/maleate polymer, or sodium silicate which is suitably present in an amount of from 1 to 5 wt%.

5 The materials that may be present in detergent compositions of the invention include sodium silicate; corrosion inhibitors including silicates; antiredeposition agents such as cellulosic polymers; fluorescers; inorganic salts such as sodium sulphate, lather control agents or lather boosters as appropriate; proteolytic and lipolytic enzymes; dyes;
10 coloured speckles; perfumes; foam controllers; and fabric softening compounds. This list is not intended to be exhaustive.

15 The base composition is suitably prepared by spray-drying a slurry of compatible heat-insensitive ingredients, and then spraying on, admixing and/or postdosing those ingredients unsuitable for processing via the slurry. The detergent particles produced according to the process of the present invention are post-dosed to the base composition by
20 conventional methods.

Detergent compositions of the invention preferably have a bulk density of at least 500 g/l, more preferably at least 550 g/litre.

25 Such powders may be prepared either by spray-drying, by post-tower densification of spray-dried powder, or by wholly non-tower methods such as dry mixing and granulation. A high-speed mixer/granulator may advantageously be used for
30 such mixing. Processes using high-speed mixer/granulators are disclosed, for example, in EP-A-340 013, EP-A-367 339, EP-A-390 251 and EP-A-420 317.

The invention is illustrated by the following non-limiting
35 Examples.

- 21 -

Examples

A VRV flash drier was used to granulate alkyl benzene
5 sulphonic acid, sodium carbonate and zeolite. The acid
precursor and carbonate were dosed in amounts equivalent to
70 wt% of neutralised sodium alkyl benzene sulphonate, in
the final product. The amount of zeolite was 25 wt% of the
final product. The remaining 5 wt% was unreacted sodium
10 carbonate, impurities and water.

An oversize fraction was separated from the granulator
output. The wt% particle size fraction of this fraction is
shown in Table 1. As can be seen, inevitably, an oversize
15 fraction also includes some intermediate size material and
dusty material.

20

Table 1
Oversize Particle Analysis
(Sieve fraction wt%)

Sieve Fraction (μm)	wt%
1400	34.1
1000	37.4
710	25.7
500	2.4
355	0.1
250	0.2
180	0
125	0.2

25

- 22 -

This oversize material was collected and fed into the mixing region of the machine, re-running the same process. The percentage of recycled oversize fraction (on basis of sodium carbonate and zeolite recycled oversize) was varied and the effect on the D50 average particle diameter. Thus, this was not a continuous recycling of oversize but the present invention also encompasses continuous recycling of oversize. The results are given in Table 2.

10

Table 2
Effect of Recycled Oversize on Particle Size

% Recycled	D50 (μm)
0	625
5	572
7.5	630
10	575
12.5	618
15	668
17.5	616
20	671
22.5	672
25	779

15

It can be seen that surprisingly, even relatively high amounts of recycled oversize particles have negligible effect on the particle size distribution of the product itself.

- 23 -

Claims

1. A process for the production of detergent particles,
5 the process comprising feeding an acid precursor of an anionic surfactant and a neutralising agent into a horizontal thin-film evaporator/drier comprising a mixing region, a drying region and a cooling region, removing the detergent particles so formed from an outlet of the
10 evaporator/drier, separating an oversize granule fraction in which at least 70 wt% of the particles have a minimum diameter of 1000 μ m from the detergent particles and recycling the oversize granule fraction to be fed back into the thin film evaporator/drier.
15
2. A process according to claim 1, wherein at least some of the recycled oversize granule fraction is fed back into the thin-film evaporator/drier before the cooling region.
- 20 3. A process according to either preceding claim, wherein at least some of the recycled oversize granule fraction is fed back into the thin-film evaporator/drier in the mixing region.
- 25 4. A process according to any preceding claim, in which the recycled oversize granule fraction constitutes an average from 1% to 50%, preferably from 5% to 25% by weight of the total solids dosed.
- 30 5. A process according to any preceding claim, wherein in the oversize granule fraction, at least 95 wt% of the particles have a minimum diameter of 700 μ m.

INTERNATIONAL SEARCH REPORT

International Application No.

PC 17 EP 01/10657

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D11/00 C11D11/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 96 06917 A (UNILEVER PLC ;UNILEVER NV (NL)) 7 March 1996 (1996-03-07) cited in the application page 1, line 4 - line 11 page 3, line 6 - line 20 page 7, line 29 - line 35 claims 1,2; example 1	1-5
X	WO 97 32002 A (UNILEVER PLC ;UNILEVER NV (NL)) 4 September 1997 (1997-09-04) cited in the application page 1, line 3 - line 9 page 3, line 29 -page 4, line 16 page 7, line 13 - line 19 page 11, line 31 -page 13, line 9 page 13, line 30 - line 32 page 21, line 1 - line 25 claims 1,2,8	1-5



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

- 'A' document defining the general state of the art which is not considered to be of particular relevance
- 'E' earlier document but published on or after the international filing date
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- 'P' document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

G document member of the same patent family

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Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Diebold, A

INTERNATIONAL SEARCH REPORT

Int. Patent Application No.
PC, EP 01/10657

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	EP 0 555 622 A (PROCTER & GAMBLE) 18 August 1993 (1993-08-18) cited in the application page 2, line 44 - line 46 page 5, line 30 - line 40 claim 1 -----	1-5

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